APPENDIX C4
WASTE CHARACTERIZATION SAMPLING METHODS

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C4-1 Headspace-Gas Sampling

C4-1a Method Requirements

All sampling must be accomplished within a radiation containment area (e.g., glovebox or hot/warm cell). The configuration of the containment area and remote-handling equipment at each sampling facility are expected to differ. A description of the containment area and remote-handling equipment must be provided in the site quality assurance project plan (QAPjP). Headspace-gas samples will be analyzed for the analytes listed in Table C8-2 of Appendix C8.

Sites may collect samples in SUMMA® canisters using the headspace gas sampling methods described in the Methods Manual. As an alternative, sites may use on-line integrated sampling/analysis systems. In this case, samples are immediately directed to an analytical instrument instead of being collected in SUMMA® canisters. The same sampling manifold and sampling heads are used with on-line integrated sampling/analysis systems and all of the requirements associated with sampling manifolds and sampling heads must be met. However, when using an on-line integrated sampling/analysis system, the sampling batch and analytical batch quality control (QC) samples are combined as on-line batch QC samples as outlined in Section C4-1b.

Manifold

This headspace gas sampling protocol employs a multiport manifold capable of collecting multiple simultaneous headspace samples for analysis and QC purposes. The manifold can be used to collect samples in SUMMA® canisters or as part of an on-line integrated sampling/analysis system. The sampling equipment must be leak checked and cleaned prior to first use and as needed thereafter. The manifold and sample canisters must be evacuated to 0.0039 inches (in.) (0.10 millimeters [mm]) mercury (Hg) prior to sample collection. Cleaned and evacuated sample canisters must be attached to the evacuated manifold before the manifold inlet valve is opened. The manifold inlet valve must be attached to a changeable filter connected to different sampling head(s), capable of punching through the metal lid of the drum or penetrating a carbon-composite filter.

The manifold must also be equipped with a purge assembly that allows applicable QC samples to be collected through the entire manifold, from the needle tip through all of the same manifold components that the drum headspace gas passes through. Field blanks shall be samples of room air collected in the sampling area in the immediate vicinity of the waste container to be sampled. If using SUMMA® canisters, field blanks are collected directly into the canister, without the use of the manifold.

The manifold, the associated sampling heads, and the headspace-gas sample volume requirements must be designed to ensure that a representative sample is collected. The manifold internal volume must be calculated and documented in the field logbook. The total volume of headspace gases collected during each sampling operation can be determined by adding the combined volume of the canisters attached to the manifold to the internal volume of the manifold. When an estimate of the available headspace gas volume can be made, less than 10 percent of that volume should be withdrawn.

As illustrated in Figure C4-1, the sampling manifold must consist of a sample side and a standard side. The dotted line indicates how the sample side shall be connected to the standard side for cleaning and collecting equipment blanks and field reference standards. The sample side must consist of the following major components:

- An applicable sampling head that forms a leak-tight connection with the headspace sampling manifold.

- A flexible hose that allows movement of the sampling head from the purge assembly (standard side) to the waste container.

- A pressure sensor(s) that must be pneumatically connected to the manifold. This manifold pressure sensor(s) must be able to measure absolute pressure in the range from 0.002 in. (0.05 mm) Hg to 39.3 in. (1,000 mm) Hg. Resolution must be ±0.0002 in. (0.005 mm) Hg at 0.0020 in. (0.05 mm) of Hg. The manifold pressure sensor(s) must have an operating range from approximately 59°F (15°C) to 104°F (40°C).

- Ports for attaching sample canisters. If using canister-based sampling methods, a sufficient number of ports must be available to allow simultaneous collection of headspace-gas samples and duplicates for volatile organic compounds (VOC) analyses. If using an on-line integrated sampling/analysis system, only one port is necessary for the collection of comparison samples. Ports not occupied with sample canisters during cleaning or headspace-gas sampling activities require a plug to prevent ambient air from entering the system. In place of using plugs, sites may choose to install valves that can be closed to prevent intrusion of ambient air into the manifold. Ports must have VCR® fittings for connection to the sample canister(s) to prevent degradation of the fittings on the canisters and manifold.

- Sample canisters, as illustrated in Figure C4-2, that are leak-free, welded stainless steel pressure vessels, with a chromium-nickel oxide (Cr-NiO) SUMMA®-passivated interior surface, bellows valve, and a pressure/vacuum gauge. All sample canisters must have VCR® fittings for connection to sampling and analytical equipment. The pressure/vacuum gauge must be mounted on each canister. It must be helium-leak tested to 1.5 x 10^{-7} standard cubic centimeters per second (cc/s), have all stainless steel construction, and be capable of tolerating temperatures to 125°C. The gauge range must be capable of indicating leaks and sample collection.

- A dry vacuum pump with the ability to reduce the pressure in the manifold to 0.05 mm Hg. A vacuum pump that requires oil may be used, but precautions must be taken to prevent diffusion of oil vapors back to the manifold. Precautions may include the use of a molecular sieve and a cryogenic trap in series between the headspace sampling ports and the pump.

- A minimum distance between the tip of the needle and the valve that isolates the pump from the manifold in order to minimize the dead volume in the manifold. The outer diameter of the system's tubing must be 1/8 in. (3.1750 mm).

- If real-time blanks are not available, the manifold must be equipped with an organic vapor analyzer (OVA) that is capable of detecting all analytes listed in Table C8-2 of Appendix C8. The OVA must be capable of measuring total VOC concentrations as low as 0.1 parts per million (ppm). Detection of 1,1,2-trichloro-1,2,2-trifluoroethane may not be possible if a photoionization detector is used. The OVA measurement must be confirmed by the collection of equipment blanks at the frequency specified in Section C4-1 to check for manifold cleanliness.

The standard side must consist of the following major elements:

- A cylinder of compressed zero air, helium, nitrogen or hydrocarbon and carbon dioxide (CO_2)-free air to clean the manifold between samples and to provide gas for the collection of equipment blanks or on-line blanks. These high-purity gases must be certified by the manufacturer to contain less than one ppm total VOCs. The gases must be metered into the standard side of the manifold by two-stage stainless steel regulators. Alternatively, a zero air generator may be used, provided a sample of the zero air is collected and demonstrated to contain less than one ppm total VOCs. Zero air from a generator must be humidified.

- Cylinders of field-reference standard gases or on-line control sample gases. These cylinders provide gases for evaluating the accuracy of the headspace-gas sampling process. Each cylinder of field-reference gas or on-line control sample gas must have a flow-regulating device. The field-reference standard gases or on-line control sample gas must be certified by the manufacturer to contain known analytes at known concentrations.

- If using an analytical method other than Fourier Transform Infrared System (FTIRS) a humidifier filled with American Society for Testing and Materials (ASTM) Type II water, connected, and opened to the standard side of the manifold between the compressed gas cylinders and the purge assembly. Dry gases flowing to the purge assembly will pick up moisture from the humidifier. Moisture is added to the dry gases to condition the equipment blanks and field-reference standards and to assist with system cleaning between headspace-gas sample collection. If using FTIRS for analysis, the sample and sampling system must be kept dry.

NOTE: Caution should be exercised to isolate the humidifier during the evacuation of the system to prevent flooding the manifold. In lieu of the humidifier, the compressed gas cylinders (e.g., zero air and field-reference standard gas) may contain water vapor in the concentration range of 1,000 to 10,000 parts per million by volume (ppmv).

- A purge assembly that allows the sampling head (sample side) to be connected to the standard side of the manifold. The ability to make this connection is required to transfer gases from the compressed gas cylinders to the canisters or on-line analytical instrument. This connection is also required for system cleaning.

- A flow-indicating device that is connected downstream of the purge assembly to monitor the flow rate of gases through the purge assembly. The flow rate through the purge assembly must be monitored to assure that excess flow exists during cleaning activities and during QC sample collection. Maintaining excess flow will prevent ambient air from contaminating the QC samples and allow samples of gas from the compressed gas cylinders to be collected near ambient pressure.

In addition to a manifold consisting of a sample side and a standard side, the area in which the manifold is operated must contain sensors for measuring ambient pressure and ambient temperature, as follows:

- The ambient-pressure sensor must have a sufficient measurement range for the ambient barometric pressures expected at the sampling location. It must be kept in the sampling area during sampling operations. Its resolution must be 1.0 mm Hg or less, and calibration must be based on National Institute of Standards and Technology (NIST), or equivalent, standards.

- The temperature sensor must have a sufficient measurement range for the ambient temperatures expected at the sampling location. The temperature sensor calibration must be traceable to NIST, or equivalent, standards.

Direct Canister

This headspace gas sampling protocol employs a canister-sampling system to collect headspace-gas samples for analysis and QC purposes without the use of the manifold described above. Rather than attaching sampling heads to a manifold, in this method the sampling heads are attached directly to an evacuated sample canister as shown in Figure C4-3.

Canisters must be evacuated to 0.0039 in. (0.10 mm) Hg prior to use and attached to a changeable filter connected to the appropriate sampling head. The sampling head(s) must be capable of punching through the metal lid of the drums and the rigid 90-mil poly liner or penetrating a carbon-composite filter to obtain the drum headspace samples. Field duplicates must be collected at the same time, in the same manner, and using the same type of sampling apparatus as used for headspace-gas sample collection. Field blanks must be samples of room air collected in the immediate vicinity of the waste-drum sampling area prior to removal of the drum lid. Equipment blanks and field-reference standards must be collected using a purge assembly equivalent to the standard side of the manifold described above. These samples must be collected from the needle tip through the same components (e.g., needle and filter) that the headspace-gas samples pass through.

The sample canisters, associated sampling heads, and the headspace-sample volume requirements ensure that a representative sample is collected. When an estimate of the available headspace-gas volume can be made, less than 10 percent of that volume should be withdrawn. A determination of the sampling head internal volume must be made and documented. The total volume of headspace gases collected during each headspace gas sampling operation can be determined by adding the volume of the sample canister(s) attached to the sampling head to the internal volume of the sampling head. Every effort must be made to minimize the internal volume of sampling heads.

Each sample canister used with the direct canister method must have a pressure/vacuum gauge capable of indicating leaks and sample collection. Canister gauges are intended to be gross leak-detection devices not vacuum-certification devices. If a canister pressure/vacuum gauge indicates an unexpected pressure change, determine if the change is a result of ambient temperature and pressure differences or a canister leak. Prior to sampling, canisters must be evacuated to 0.0039 in. (0.10 mm) Hg. This gauge must be helium-leak tested to 1.5×10^{-7} standard cc/s, have all stainless steel construction, and be capable of tolerating temperatures to 125°C.

The SUMMA® sample canisters must be used when sampling each drum. Three different sampling heads for attachment to the sample canister are described below. These heads must form a leak-tight connection with the canister and allow sampling through the drum-lid carbon-composite filter, or through the drum lid itself. Figure C4-3 illustrates the direct canister-sampling equipment.

<u>Sampling Heads</u>

A sample of the headspace gas directly under the drum lid must be collected from within the drum. Two methods, sampling through the carbon filter and sampling through the drum lid, have been developed for collecting a representative sample.

<u>Sampling Through the Carbon Filter</u>

To sample the drum-headspace gas through the drum's carbon-composite filter, a side-port needle (i.e., a hollow needle sealed at the tip with a small opening on its side close to the tip) must be pressed through the filter and into the headspace beneath the drum lid. This permits the gas to be drawn into the manifold or directly into the canister(s). This procedure is described in detail in the Methods Manual and is specific to a type of carbon-composite filter that permits insertion of the needle. To assure that the sample collected is representative, all of the general method requirements, sampling apparatus requirements, and QC requirements described in this section must be met in addition to the following requirements that are pertinent to drum headspace-gas sampling through the carbon filter:

- The lid of the drum's 90-mil poly liner must contain a hole for venting to the drum. If headspace-gas samples are collected prior to venting the 90-mil poly liner, a nonconformance report must be prepared, submitted, and resolved. Nonconformance procedures are outlined in Appendix C8 (Section C8-13).

- For sample collection, the drum's carbon-composite filter must be sealed as specified in the Methods Manual, or equivalent, to prevent outside air from entering the drum and diluting and/or contaminating the sample.

The sampling head for collecting drum headspace by penetrating the carbon-composite filter must consist of a side-port needle, a filter to prevent particles from contaminating the gas sample, and an adapter to connect the two. To prevent cross contamination, the sampling head must be cleaned or replaced after sample collection, after field-reference standard collection, and after field-blank collection. The following requirements must also be met:

- The housing of the carbon-composite filter must allow insertion of the sampling needle through the filter element into the drum headspace.

- The side-port needle must be used to reduce the potential for plugging.

- The purge assembly must be modified for compatibility with the side-port needle.

<u>Sampling Through the Drum Lid</u>

Sampling through the drum lid must be performed when the drum's carbon-composite filter does not permit insertion of the side-port needle. To sample the drum headspace-gas through the drum lid, the lid must be breached using a sparkless punch. The punch must form an airtight seal between the drum lid and the manifold or direct canister. To assure that the sample collected is representative, all of the general method requirements, sampling apparatus requirements, and QC requirements specified in Methods Manual Procedures 110.1 through 110.4, as appropriate, must be met in addition to the following requirements:

- The seal between the drum lid and sampling head must be designed to minimize intrusion of ambient air [See Methods Manual Procedure 110.4, Section 8.0].

- All components of the drum-punch sampling system that come into contact with sample gases must be purged with humidified zero air, nitrogen, or helium prior to sample collection [See Methods Manual Procedure 110.4, Section 8.0].

- Equipment blanks and field reference standards must be collected through all the components of the punch that contact the headspace-gas sample.

- Pressure must be applied to the sparkless punch until the drum lid has been breached. Then the punch must be backed out to expose the headspace gas.

- Provisions must be made to relieve potential drum pressure increases during drum-punch operations; pressure increases may occur during sealing of the drum punch to the drum lid.

- The lid of the drum's 90-mil poly liner must contain a hole for venting to the drum. If headspace-gas samples are collected prior to venting the 90-mil poly liner, a nonconformance report must be prepared, submitted, and resolved.

- During sampling, the drum's carbon-composite filter, if present, must be sealed to prevent outside air from entering the drum.

Sampling through the drum lid must be accomplished using the drum punch described in the Methods Manual (Procedure 110.4), or an equivalent. The same type of sampling head as used for the 55-gallon (208-liter) poly bag sampling must be pneumatically connected to the drum punch to provide a seal between the drum lid and the manifold or direct canister. The following requirements must also be met:

- A flow-indicating device to verify excess flow of QC gases (for system purge) must be pneumatically connected downstream of the drum punch and operated in the same manner as the flow-indicating device described in the "Manifold" section. A flowrate of approximately one liter per minute for approximately three minutes is required.

- Equipment must be used adequately to secure the drum-punch sampling system to the drum lid.

- Provisions must be made to prevent the punch from rotating as it is pressed through the drum lid.

C4-1b <u>Quality Control</u>

For manifold and direct canister sampling systems, field QC samples must be collected on a per sampling batch basis. A sampling batch is a suite of samples collected consecutively using the same sampling equipment within a specific time period. A sampling batch can be up to 20 samples (excluding QC samples), all of which must be collected within 14 days of the first sample in the batch. For on-line integrated sampling/analysis systems, QC samples must be collected and analyzed on a per on-line batch basis. An on-line batch is the number of headspace gas samples collected and analyzed within a 12-hour period using the same on-line integrated analysis system. Table C4-2 provides a summary of field QC sample collection requirements. Table C4-3 provides a summary of QC sample acceptance criteria.

For on-line integrated sampling analysis systems, the on-line batch QC samples serve as combined sampling batch/analytical batch QC samples as follows:

- The on-line blank replaces the equipment blank and laboratory blank

- The on-line control sample replaces the field reference standard and laboratory control sample

- The on-line duplicate replaces the field duplicate and laboratory duplicate

The acceptance criteria for on-line batch QC samples are the same as for the sampling batch and analytical batch QC samples they replace. Acceptance criteria are shown in Table C4-3. A separate field blank must still be collected and analyzed for each on-line batch. However, if the results of a field blank collected through the sampling manifold meets the acceptance criterion, a separate on-line blank need not be collected and analyzed.

The site project Quality Assurance (QA) officer shall have the responsibility to monitor and document field QC sample results and fill out a nonconformance report if acceptance criteria are not met. The site project manager shall have the responsibility to ensure appropriate corrective action is taken if acceptance criteria are not met.

Field Blanks

Field blanks must be collected to evaluate background levels of program-required analytes. Field blanks must be collected prior to sample collection, and at a frequency of one per sampling batch. The site project manager shall use the field blank data to assess impacts of ambient contamination, if any, on the sample results. A nonconformance report (Section C8-13) must be initiated and resolved if the final reported QC sample results do not meet the acceptance criteria.

Equipment Blanks

Equipment blanks must be collected to assess cleanliness prior to first use of all sampling equipment. After the initial cleanliness check, equipment blanks collected through the manifold must be collected at a frequency of one per sampling batch for VOC analysis. If the direct canister method is used, field blanks may be used in lieu of equipment blanks. The site project manager shall use the equipment blank data to assess impacts of potentially contaminated sampling equipment on the sample results. Equipment blank results determined by gas chromatography/mass spectrometry and gas chromatography/flame ionization detection shall be acceptable if the concentration of each VOC analyte is less than three times the method detection limit (MDL) listed in Table C8-2 in Appendix C8. Equipment blank results determined by FTIRS shall be acceptable if the concentration of each VOC analyte is less than the program required quantitation limit and listed in Table C8-2.

Field Reference Standards

Field reference standards shall be used to assess the accuracy with which the sampling equipment collects VOC samples into SUMMA® canisters prior to first use of the sampling equipment. Field reference standards must contain a minimum of six of the analytes listed in Table C8-2 in Appendix C8 at concentrations within a range of 0 to 100 ppmv. Field reference standards must have a known valid relationship to a nationally recognized standard (e.g., NIST). If commercial gases are used, a Certificate of Analysis from the manufacturer documenting traceability is required. Commercial stock gases must not be used beyond their manufacturer-specified shelf life. After the initial accuracy check, field reference standards collected through the manifold must be collected at a frequency of one per sampling batch and submitted blind to the analytical laboratory. For the direct canister method, field reference standard collection may be discontinued if the field reference standard results demonstrate the quality assurance objectives (QAO) for accuracy specified in Appendix C8. Field reference standard results shall be acceptable if the accuracy is 70 to 130 percent recovery.

Field Duplicates

Field duplicate samples must be collected simultaneously and in accordance with Table C4-1 to assess the precision with which the sampling procedure can collect samples into SUMMA® canisters. Field duplicate results shall be acceptable if the relative percent difference is less than or equal to 25.

C4-1c Equipment Testing, Inspection and Maintenance

All sampling equipment components that come into contact with headspace sample gases must be constructed of relatively inert materials such as stainless steel or Teflon®. A passivated interior surface on the stainless steel components is recommended.

To minimize the potential for cross contamination of samples, the headspace sampling manifold and sample canisters must be properly cleaned and leak-checked prior to headspace gas sampling. Procedures for cleaning and preparing the manifold and sample canisters are provided in the Methods Manual (Procedures 110.1 and 110.2). Cleaning requirements are presented below.

Headspace Gas Sample Canister Cleaning

SUMMA® canisters used in these methods must be subjected to a rigorous cleaning and certification procedure prior to use in the collection of any samples. Guidance for the development of this procedure has been derived from Method TO-14 (EPA 1988a) and can be found in the Methods Manual (Procedure 210.1). Specific details must be provided in laboratory standard operating procedures (SOPs) for the cleaning and certification of canisters.

Canisters must be cleaned and certified on an equipment cleaning batch basis. An equipment cleaning batch is any number of canisters cleaned together at one time using the same cleaning method. A cleaning system, capable of processing multiple canisters at a time, composed of an oven (optional) and a cryogenic trap vacuum manifold must be used to clean SUMMA® canisters. Prior to cleaning, a 24-hour leak test must be performed on all canisters. For a positive pressure check, a canister passes if the pressure does not change by more than ±2 psig in 24 hours. Any canister that fails must be checked for leaks, repaired, and reprocessed. One canister per equipment cleaning batch must be filled with humid zero air or humid high purity nitrogen and analyzed for VOCs. The equipment cleaning batch of canisters shall be considered clean if there are no VOCs above three times the MDLs listed in Table C8-2 of Appendix C8. After the canisters have been certified for leak-tightness and free of background contamination, they must be evacuated to 0.0039 in. (0.10 mm) Hg or less for storage prior to shipment. The laboratory responsible for canister cleaning and certification shall maintain canister certification documentation and initiate the canister tags as described in Section 6.0 of the Transuranic Waste Characterization Quality Assurance Program Plan (QAPP).

Sampling Equipment Initial Cleaning and Leak Check

The surfaces of all headspace gas sampling equipment components that will come into contact with headspace gas must be thoroughly inspected and cleaned prior to assembly, in accordance with Methods Manual Procedures 110.1 and 110.2, or equivalent. The manifold and associated sampling heads must be purged with humidified zero air, nitrogen, or helium, and leak checked after assembly. This cleaning must be repeated if the manifold and/or associated sampling heads are contaminated to the extent that the routine system cleaning is inadequate.

Sampling Equipment Routine Cleaning and Leak Check

The manifold and associated sampling heads which are reused must be cleaned and checked for leaks in accordance with the cleaning and leak check procedures described in Procedures 110.1 and 110.2 of the Methods Manual, or equivalent. The procedures must be conducted after headspace gas and field duplicate collection; after field blank collection, if the field blank is collected through the manifold; and after the additional cleaning required for field reference standard collection has been completed. The protocol for routine manifold cleaning and leak check requires that sample canisters be attached to the canister ports, or that the ports be capped or closed by valves, and requires that the sampling head be attached to the purge assembly. Humidified zero air, nitrogen, or helium, regulated through the purge assembly, must then be swept through the sample side of the sampling system.

VOCs must be removed from the internal surfaces of the headspace sampling manifold to levels that are less than three times the MDLs of the analytes listed in Table C8-2 of Appendix C8, as determined by analysis of an equipment blank or the OVA. This is achieved by sweeping the sample side of the sampling system. It is recommended that the headspace sampling manifold be heated and periodically evacuated and flushed with humidified zero air, nitrogen, or helium. When not in use, the manifold must be demonstrated clean before storage with a positive pressure of high purity gas (i.e., zero air, nitrogen, or helium) in both the standard and sample sides.

Sampling must be suspended and corrective actions must be taken when the analysis of an equipment blank indicates these limits have been exceeded. The site project manager must ensure that corrective action has been taken prior to resumption of sampling.

Manifold Cleaning After Field Reference Standard Collection

The sampling system must be specially cleaned after a field reference standard has been collected, because the field reference standard gases contaminate the standard side of the headspace sampling manifold when they are regulated through the purge assembly. This cleaning requires the installation of a gas-tight connector in place of the sampling head, between the flexible hose and the purge assembly. This configuration allows both the sample and standard sides of the sampling system to be flushed (evacuated and pressurized) with humidified zero air, nitrogen, or helium which, combined with heating the pneumatic lines, should sweep and adequately clean the system's internal surfaces. After this protocol has been completed and prior to collecting another sample, the routine system cleaning and leak check (see previous section) must also be performed.

Sampling Head Cleaning

To prevent cross contamination, the needle, adapters, and filter of the sampling heads must be cleaned in accordance with the cleaning procedures described in Procedures 110.1 and 110.2 of the Methods Manual, or equivalent. After sample collection, a sampling head must be disposed of or cleaned in accordance with the Methods Manual procedures, or equivalent, prior to reuse. As a further QC measure, the needle and filter, after cleaning, should be purged with zero air, nitrogen, or helium and capped for storage to prevent sample contamination by VOCs potentially present in ambient air.

C4-1d Equipment Calibration and Frequency

The manifold pressure sensor must be certified prior to initial use, then annually, using NIST traceable, or equivalent, standards. If necessary, the pressure indicated by the pressure sensor(s) must be temperature compensated. The ambient air temperature sensor, if present, must be certified prior to initial use, then annually, to NIST traceable, or equivalent, temperature standards.

The OVA must be calibrated once per day, prior to first use, or as necessary according to the manufacturer's specifications. Calibration gases must be certified to contain known analytes at known concentrations. The balance of the OVA calibration gas must be consistent with the manifold purge gas when the OVA is used (i.e., zero air, nitrogen, or helium).

C4-2 Sampling of Homogenous Solids and Soil/Gravel

C4-2a Method Requirements

The methods used to collect samples of transuranic (TRU) waste, classified as homogenous solids and soil/gravel from waste containers, must be such that the samples are representative of the waste from which they were taken. To minimize the quantity of investigation-derived waste, laboratories conducting the analytical work may require no more sample than is required for the analysis, based on the analytical methods. Therefore, sampling must be conducted to collect samples in accordance with the QAO specifications as described below.

Core Collection

Coring tools must be used to collect cores of homogenous solids and soil/gravel from waste containers, when possible, in a manner that minimizes disturbance to the core. A rotational coring tool (i.e., a tool that is rotated longitudinally), similar to a drill bit, to cut, lift the waste cuttings, and collect a core from the bore hole, must be used to collect sample cores from containers of the waste. For homogenous solids and soil/gravel that are relatively soft, nonrotational coring tools may be used in lieu of a rotational coring tool.

To provide a basis for describing the requirements for core collection, diagrams of a rotational coring tool (i.e., a light weight auger) and a nonrotational coring tool (i.e., a thin-walled sampler) are provided in Figures C4-4 and C4-5, respectively. Each has been tested for its ability to collect a vertical core of simulated solidified waste contained in 55-gal (208-L) drums and 1-gal (3.8-L) poly bottles (EG&G 1994). The nonrotational coring tool has demonstrated core recoveries greater than 88 percent for soft simulated wastes. The rotational coring tool has demonstrated core recoveries greater than 75 percent for soft simulated wastes and greater than 94 percent for hard simulated wastes.

The following requirements apply to the use of coring tools:

- Each coring tool must contain a removable tube (liner) that is constructed of fairly rigid material unlikely to affect the composition and/or concentrations of target analytes in the sample core. Materials that are acceptable for use for coring device sleeves are polycarbonate, teflon, or glass for most samples, and stainless steel or brass if samples are not to be analyzed for metals (Methods Manual Procedure 120.1). Site QAPjPs must document that analytes of concern are not likely to be present in liner material. Sites must document that the materials are unlikely to affect sample results through the collection and analysis of equipment prior to first use as specified in the 'Equipment Blanks' section of this appendix. Liner outer diameter is recommended to be no more than 2 in. and no less than one in. Liner wall thickness is recommended to be no greater than 1/16 in. Before use, the liner must be cleaned in accordance the requirements in Section C4-2b. The liner must fit flush with the inner wall of the coring tool and must be of sufficient length to hold a core that is representative of the waste along the entire depth of the waste. The liner material must have sufficient transparency to allow visual examination of the core after sampling. If sub-sampling is not conducted immediately after core collection and liner extrusion, then end caps constructed of material unlikely to affect the composition and/or concentrations of target analytes in the core (e.g., Teflon®) must be placed over the ends of the liner. End caps must fit tightly to the ends of the liner.

- A spring retainer, similar to that illustrated in Figures C4-4 and C4-5, must be used with each coring tool when the physical properties of the waste are such that the waste may fall out of the coring tool's liner during sampling activities. The spring retainer must be constructed of relatively inert material (e.g., stainless steel or Teflon®) and its inner diameter must not be less than the inner diameter of the liner. Before use, spring retainers must be cleaned in accordance with the requirements in Section C4-2b.

- Coring tools must have an air-lock mechanism that opens to allow air inside the liners to escape as the tool is pressed into the waste (e.g., ball check valve). This air-lock mechanism must also close when the core is removed from the waste container.

- After disassembling the coring tool, a device (extruder) to forcefully extrude the liner from the coring tool must be used if the liner does not slide freely. All surfaces of the extruder that may come into contact with the core must be cleaned in accordance with the requirements in Section C4-2(b) prior to use.

- Coring tools must be of sufficient length to hold the liner and must be constructed to allow placement of the liner leading edge as close as possible to the coring tools leading edge.

- All surfaces of the coring tool that have the potential to contact the sample core must be cleaned in accordance with the requirements in Section C4-2(b) prior to use.

- The leading edge of the coring tools must be sharpened and tapered to a diameter equivalent to, or slightly smaller than, the inner diameter of the liner. Based on tests conducted with the coring tools described in the Methods Manual, a diameter slightly smaller (e.g., 1/10 in.) has demonstrated a reduction in the drag of the homogenous solids and soil/gravel against the internal surfaces of the liner, thereby enhancing sample recovery.

- Rotational coring tools must have a mechanism to prevent the liner inside the coring tool from rotating with the coring tool during coring activities, thereby minimizing physical disturbance to the core.

- Rotational coring must be conducted in a manner that minimizes transfer of frictional heat to the core, thereby minimizing potential loss of VOCs.

- Nonrotational coring tools must be designed such that the tool's kerf width is minimized. Kerf width is defined as one-half of the difference between the outer diameter of the tool and the inner diameter of the tool's inlet.

<u>Sample Collection</u>

Sampling must be conducted in accordance with the following requirements:

- Sampling must be conducted as soon as possible after core collection. If a substantial delay (i.e., more than 60 minutes) is expected between core collection and sampling, the core must remain in the liner and the liner must be capped at each end. If the liner containing the core is not extruded from the coring tool and capped, then two alternatives are permissible: 1) the liner must be left in the coring tool and the coring tool must be capped at each end, or 2) the coring tool must remain in the waste container with the air-lock mechanism attached.

- Samples of homogenous solids and soil/gravel for VOC analyses must be collected prior to extruding the core from the liner. The sampling location must be randomly selected along the long axis of the liner and access to the waste must be gained by making a perpendicular cut through the liner and the core. Sites must develop procedures to select, and document the selection, of random sampling locations. True random sampling involves the proper use of random numbers for identifying sampling locations. A sampling device such as the metal coring cylinder described in ASTM Designation: 4547-91 (ASTM 1991a), or modified disposable syringe described in Procedure 120.1 of the Methods Manual, or equivalent, must be immediately used to collect a 15-gram sample once the core has been exposed to air. Immediately after sample collection, the sample must be extruded into a 40-mL Volatile Organics Analysis (VOA) vial, the top rim of the vial visually inspected and wiped clean of any waste residue, and the vial cap secured. Sample handling requirements are outlined in Table C4-4. Additional guidance for this type of sampling can be found in *Soil Sampling and Analysis for Volatile Organic Compounds* (EPA, 1991).

- Samples of the homogenous solids and soil/gravel for semi-volatile organic compound, polychlorinated biphenyls, and metals analyses must be collected. These samples may be collected from the same location and in the same manner as the sample(s) collected for VOC analysis, or they may be collected by splitting or compositing a representative subsection of the core. The representative subsection is chosen by randomly selecting a location along the core. Sites must develop procedures to select, and document the selection, of random sampling locations. True random sampling involves the proper use of random numbers for identifying sampling locations. Guidance for splitting and compositing solid materials can be found in "Standard Practice for Reducing Field Samples of Aggregate to Testing Size" (ASTM, 1987). All surfaces of the sampling tools that have the potential to come into contact with the sample must be constructed of materials unlikely to affect the composition or concentrations of target analytes in the waste (e.g., Teflon®). Sample sizes and handling requirements are outlined in Table C4-4.

C4-2b Quality Control

QC requirements for sampling of homogenous solids and soil/gravel include collection of collocated cores to determine precision; equipment blanks to verify cleanliness of the coring tools and sampling equipment; and analysis of reagent blanks to ensure reagents, such as deionized or high pressure liquid chromatography (HPLC) water, are of sufficient quality. Coring and sampling of homogenous solids and soil/gravel must comply, at minimum, with the following QC requirements.

Co-located Cores

In accordance with the requirement to collect field duplicates required by Environmental Protection Agency (EPA) methods found in SW-846, co-located cores must be collected to determine the combined precision of the coring and sampling procedures. The co-located core methodology is a duplicate sample collection methodology intended to collect samples from approximately the same location within the drum. Cores must be collected side by side as close as feasible to one another, handled in the same manner, visually inspected through the transparent liner, and sampled in the same manner at the same randomly selected sample location. If the visual examination detects inconsistencies such as color, texture, or waste type in the waste at the sample location, another sampling location may be randomly selected, or the cores may be invalidated and co-located cores may again be collected. Co-located cores must be collected at a frequency of one per sampling batch. A sampling batch is a suite of homogenous solids and soil/gravel samples collected consecutively using the same sampling equipment within a specific time period. A sampling batch can be up to 20 samples (excluding field QC samples), all of which must be collected within 14 days of the first sample in the batch. Because of the normally slow rate of core collection (1-2 cores per day), daily collection of field QC samples would result in numerous QC samples being collected for each field sample. This is inappropriate for sampling operations and is unnecessary for QC purposes. The collection of field QC samples on a "per sampling batch" basis provides adequate control for sampling operations.

Equipment Blanks

In accordance with SW-846, equipment blanks must be collected from fully assembled coring tools prior to first use at a frequency of one per equipment cleaning batch. An equipment cleaning batch is the number of sampling equipment items cleaned together at one time using the same cleaning method. The equipment blank must be collected from the fully assembled coring tool, in the area where the coring tools are cleaned, prior to covering with protective wrapping and storage. The equipment blank must be collected by pouring clean water (e.g., deionized water, HPLC water) down the inside of the liners of the assembled coring tool. The water must be collected in a clean sample container placed at the leading edge of the coring tool. and analyzed for the analytes listed in Tables C8-4, C8-6, and C8-9 of Appendix C8. The results of the equipment blank will be considered acceptable if the analysis indicates no analyte at a concentration greater than three times the MDLs listed in Tables C8-4 and C8-6 or in the Program Required Detection Limits (PRDL) in Table C8-9 of Appendix C8. If analytes are detected at concentrations greater than three times the MDLs, then the associated equipment cleaning batch of coring tools must be cleaned again and another equipment blank collected.

Equipment blanks must be collected from liners that are cleaned separately from the coring tools. These equipment blanks must be collected at a frequency of one per equipment cleaning batch. The equipment blanks must be collected by randomly selecting a liner from the equipment cleaning batch, pouring clean water (e.g., deionized water or HPLC water) across its internal surface, collecting the water in a clean sample container, and analyzing the water for the analytes listed in Tables C8-4, C8-6, and the PRDLs in C8-9 of Appendix C8. The results of the equipment blank analysis will be considered acceptable if the results indicate no analyte at a concentration greater than three times the MDLs listed in Tables C8-4, C8-6, or C8-9 of Appendix C8. If analytes are detected at concentrations greater than three times the MDLs (or PRDLs for metals), then the associated equipment cleaning batch of liners must be cleaned again and another equipment blank collected.

Sampling equipment (e.g., bowls, spoons, chisel, VOC sub-sampler) must also be cleaned. Equipment blanks must be collected for the sampling equipment at a frequency of one per equipment cleaning batch. After the sampling equipment has been cleaned, one item from the equipment cleaning batch is randomly selected, water (e.g., deionized water, HPLC water) is passed over its surface, collected in a clean container, and analyzed for the analytes listed in Tables C8-4, C8-6, and C8-9 of Appendix C8. The results of the equipment blank will be considered acceptable if the results indicate no analyte present at a concentration greater than three times the MDLs listed in Tables C8-4 and C8-6 and in the PRDLs in C8-9 of Appendix C8. If analytes are detected at concentrations greater than three times the MDLs (or PRDLs for metals), then the associated equipment cleaning batch of sampling equipment must be cleaned again and another equipment blank collected.

The results of equipment blanks must be traceable to the items in the equipment cleaning batch that the equipment blank represents. It is recommended that the equipment blank results for the coring tools, liners, and sampling equipment be reviewed prior to use. A sufficient quantity of these items should be maintained in storage to prevent disruption of sampling operations.

A site may choose to discard liners and sampling tools after one use. In this instance, cleaning and equipment blank collection is not required.

Coring Tool and Sampling Equipment Cleaning

Coring tools and sampling equipment must be cleaned in accordance with the following requirements:

- All surfaces of coring tools and sampling equipment that will come into contact with the core and the samples must be clean prior to use. All items of sampling equipment must be cleaned in the same manner. Immediately following cleaning, coring tools and sampling equipment must be assembled and sealed inside clean protective wrapping.

- Each coring tool must have a unique identification number. Each number must be referenced to the waste container on which it was used. This information must be recorded in the field records. One coring tool from the equipment cleaning batch must be tested for cleanliness in accordance with the requirements specified above. The identification number of the coring tool from which the equipment blank was collected must be recorded in the field records. The results of the equipment blank analysis for the equipment cleaning batch in which each coring tool was cleaned must be submitted to the sampling facility with the identification numbers of all coring tools in the equipment cleaning batch.

- Sample containers must be cleaned in accordance with the *Specifications and Guidance for Obtaining Contaminant-Free Sample Containers* (EPA, 1992).

C4-2c Equipment Testing, Inspection and Maintenance

Prior to initiation of coring activities, coring tools must be tested in accordance with manufacturer specifications to ensure operation within the manufacturer's tolerance limits. Other specifications specific to the sampling operations (e.g., operation of containment structure and safety systems) should also be tested and verified as operating properly prior to initiating coring activities. Coring tools must be assembled, including liners, and tested. Air-lock mechanisms and rotation mechanisms must be inspected for free movement of critical parts. Coring tools found to be malfunctioning must be repaired or replaced prior to use.

Coring tools and sample collection equipment must be maintained in accordance with manufacturer's specifications. Clean coring tools and sampling equipment must be sealed inside clean protective wrapping and maintained in a clean storage area prior to use. Sampling equipment must be properly maintained to avoid contamination. A sufficient supply of spare parts should be maintained to prevent delays in sampling activities due to equipment down time. Records of equipment maintenance and repair must be maintained in the field records in accordance with site SOPs.

Inspection of sampling equipment and work areas shall include the following:

- Sample collection equipment in the immediate area of sample collection must be inspected daily for cleanliness. Visible contamination on any equipment (e.g., waste on floor of sampling area, hydraulic fluid from hoses) that has the potential to contaminate a waste core or waste sample must be thoroughly cleaned upon its discovery.

- The waste coring and sampling work areas must be maintained in clean condition to minimize the potential for cross contamination between cores and samples.

- Expendable equipment (e.g., plastic sheeting, plastic gloves) must be visually inspected for cleanliness prior to use and properly discarded after each sample.

- Prior to removal of the protective wrapping from a coring tool designated for use, the condition of the protective wrapping must be visually assessed. Coring tools with torn protective wrapping should be returned for cleaning. Coring tools visibly contaminated after the protective wrapping has been removed must not be used and must be returned for cleaning or properly discarded.

- Sampling equipment must be visually inspected prior to use. All sampling equipment that comes into contact with waste samples must be stored in protective wrapping until use. Prior to removal of the protective wrapping from sampling equipment, the condition of the protective wrapping must be visually assessed. Sampling equipment with torn protective wrapping should be discarded or returned for cleaning. Sampling equipment visibly contaminated after the protective wrapping has been removed must not be used and must be returned for cleaning or properly discarded.

C4-2d <u>Equipment Calibration and Frequency</u>

The scale used for weighing sub-samples must be calibrated as necessary to maintain its operation within manufacturer's specification, and after repairs and routine maintenance. Weights used for calibration must be traceable to a nationally recognized standard. Calibration records must be maintained in the field records.

C4-3 Radiography

C4-3a Methods Requirements

Radiography has been developed by the Department of Energy (DOE) specifically to aid in the examination and identification of containerized waste. There is no equivalent or associated method found in EPA sampling and analysis guidance documents. All activities required to achieve the radiography objectives must be described in site QAPjPs and SOPs.

A radiography system normally consists of an X-ray-producing device, an imaging system, an enclosure for radiation protection, a waste container handling system, an audio/video recording system, and an operator control and data acquisition station. Although these six components are required, it is expected there will be some variation within a given component between sites. The X-ray-producing device must have controls which allow the operator to vary the voltage, thereby controlling image quality. It should be possible to vary the voltage, typically between 150 to 400 kilovolts (k), to provide an optimum degree of penetration through the waste. For example, high-density material should be examined with the X-ray device set on the maximum voltage. This ensures maximum penetration through the waste container. Low-density material should be examined at lower voltage settings to improve contrast and image definition. The imaging system typically utilizes a fluorescent screen and a low-light television camera.

To perform radiography, the waste container is scanned while the operator views the television screen. An audio/videotape is made of the waste container scan and is maintained as a permanent record. A radiography data form is also used to document the matrix parameter category and estimated waste material parameter weights of the waste. The estimated waste material parameter weights should be determined by compiling an inventory of waste items, residual materials, and packaging materials. The items on this inventory should be sorted by waste material parameter and combined with a standard weight look-up table to provide an estimate of waste material parameter weights.

C4-3b Quality Control

The radiography system involves qualitative and semiquantitative evaluations of visual displays. Operator training and experience are the most important considerations for assuring quality controls in regard to the operation of the radiography system and for interpretation and disposition of radiography results. Only trained personnel must be allowed to operate radiography equipment.

Standardized training requirements for radiography operators must be based upon existing industry standard training requirements and must comply with the training and qualification requirements of NQA-1, Element 2, except for Supplement 2S-2 (ASME, 1994). Supplement 2S-2 is associated with radiography used in verifying safety-related parameters, such as welding, where quantitative comparisons can be utilized. As such, it is not applicable to waste management operations and not considered necessary or appropriate for training radiography operators involved in TRU waste characterization activities.

Each site must develop a training program that provides radiography operators with both formal and on-the-job (OJT) training. Radiography operators must be instructed in the specific waste generating practices, typical packaging configurations, and associated waste material parameters expected to be found in each matrix parameter category at the site. The OJT and apprenticeship must be conducted by an experienced, qualified radiography operator prior to qualification of the training candidate. The training programs will be site-specific due to differences in equipment, waste configurations, and the level of waste characterization efforts. For example, certain sites use digital radiography equipment, which is more sensitive than real-time radiography equipment. In addition, the particular physical forms and packaging configurations at each site will vary; therefore, radiography operators must be trained on the types of waste that are generated, stored, and/or characterized at that particular site.

Although each site must develop its own training program, all of the radiography QC requirements specified in this Waste Analysis Plan (WAP) and the Methods Manual must be incorporated into the training programs and radiography operations. In this way data quality and comparability will not be affected.

Radiography training programs will be the subject of the Generator/Storage Site Waste Screening and Acceptance Audit Program (Appendix C11).

Although the site-specific training programs will vary to some degree, each program will contain the following required elements based on NQA-1 requirements:

Formal Training

- Project Requirements
- State and Federal Regulations
- Basic Principles of Radiography
- Radiographic Image Quality
- Radiographic Scanning Techniques
- Application Techniques
- Radiography of Waste Forms
- Standards, Codes, and Procedures for Radiography
- Site-Specific Instruction

On-the-Job Training

- System Operation
- Identification of Packaging Configurations
- Identification of Waste Material Parameters
- Weight and Volume Estimation
- Identification of Prohibited Items

A radiography test drum will include items common to the waste streams to be generated/stored at the generator/storage site. The test drums must be divided into layers with varying packing densities or different drums may be used to represent different situations that may occur during radiography examination at the site. The following is a list of required elements of a radiography test drum:

- Aerosol can with puncture
- Horsetail bag
- Pair of coveralls
- Empty bottle
- Irregular shaped pieces of wood
- Empty one gallon paint can
- Full container
- Aerosol can with fluid
- One gallon bottle with three tablespoons of fluid
- One gallon bottle with one cup of fluid (upside down)
- Leaded glove or leaded apron
- Wrench

These items must be successfully identified by the operator as part of the qualification process. Qualification of radiography operators must, at a minimum, encompass the following requirements:

- Successfully pass a comprehensive exam based upon training enabling objectives. This exam will be reviewed as part of the Generator/Storage Site Waste Screening and Acceptance Audit Program (Appendix C11)

- Perform practical capability demonstration in the presence of appointed site radiography subject matter expert. This person is an experienced radiography operator who is qualified as an OJT trainer.

Requalification of operators must be based upon evidence of continued satisfactory performance (primarily audio/videotape reviews) and must be done at least every two years. Unsatisfactory performance will result in disqualification. Unsatisfactory performance is defined as the misidentification of a prohibited item in a training drum or a score of less than 80% on the comprehensive exam. Retraining and demonstration of satisfactory performance are required before an operator is again allowed to operate the radiography system.

A training drum with various container sizes must be periodically scanned by each operator. The videotape must then be reviewed by a supervisor to ensure that operators' interpretations remain consistent and accurate. Imaging system characteristics must be verified on a routine basis.

Independent replicate scans and replicate observations of the video output of the radiography process must be performed under uniform conditions and procedures. Independent replicate scans must be performed on one waste container per day or once per testing batch, whichever is less frequent. Independent observations of one scan (not the replicate scan) must also be made once per day or once per testing batch, whichever is less frequent, by a qualified radiography operator other than the individual who performed the first examination. A testing batch is a suite of waste containers undergoing radiography using the same testing equipment A testing batch can be up to 20 waste containers without regard to waste matrix.

Oversight functions include periodic audio/video tape reviews of accepted waste containers and must be performed by qualified radiography personnel other than the operator who dispositioned the waste container. The results of this verification must be available to the radiography operator. The site project QA officer shall be responsible for monitoring the quality of the radiography data and calling for corrective action, when necessary.

Visual Examination

As an additional QC check, the radiography results must be verified directly by visual examination of the waste container contents. Visual examination must be performed on a statistically determined portion of waste containers to verify the results of radiography. This verification must include the matrix parameter category and waste material parameter weights. The verification must be performed through a comparison of radiography and visual examination results. The results of the visual examination must be transmitted to the radiography facility.

The visual examination must consist of a semi-quantitative and/or qualitative evaluation of the waste container contents, and must be recorded on audio/videotape. The visual examination program has been developed by the DOE to provide an acceptable level of confidence in radiography. There is no equivalent method found in EPA sampling and analysis guidance documents. A detailed procedure that meets the requirements of this method can be found in the Methods Manual.

Standardized training for visual inspection must be developed to include both formal classroom and OJT. Visual inspectors must be instructed in the specific waste generating processes, typical packaging configurations, and expected waste material parameters expected to be found in each matrix parameter category at the site. The OJT and apprenticeship must be conducted by an operator experienced and qualified in visual examination prior to qualification of the candidate. The training must be site specific to include the various waste configurations generated/stored at the site. For example, the particular physical forms and packaging configurations at each site will vary so operators must be trained on types of waste that are generated, stored, and/or characterized at that particular site. Visual examination personnel must be requalified once every two years.

Although site-specific training programs will vary to some degree, each program will contain the following required elements based on NQA-1 requirements:

Formal Training

- Project Requirements
- State and Federal Regulations
- Application Techniques
- Site-Specific Instruction

On-the-Job Training

- Identification of Packaging Configurations
- Identification of Waste Material Parameters

- Weight and Volume Estimation
- Identification of Prohibited Items

Each visual examination facility must designate a visual examination expert. The visual examination expert must be familiar with the waste generating processes that have taken place at that site and also be familiar with all of the types of waste being characterized at that site. The visual examination expert shall be responsible for the overall direction and implementation of the visual examination at that facility. Site QAPjPs must specify the selection, qualification, and training requirements of the visual examination expert.

Figure C4-6 illustrates the overall programmatic approach to the visual examination of waste. If the waste is homogeneous, the expert may decide that a limited visual examination involving a confirmation of the radiography data is appropriate. If the waste is heterogeneous, the expert may decide a full visual examination by opening bags and segregating waste is warranted. Various degrees of segregation are possible based on the expert's judgment and availability of acceptable knowledge data. Site QAPjPs must specify decision-making criteria for the visual examination expert. In all cases, SOPs must be developed to support the visual examination process, and the basis for the expert's decisions must be documented.

A description of the waste container contents must be recorded on a data form as implemented in the site QAPjP. The description can be brief, but it must clearly identify the appropriate waste matrix parameters and provide enough information to estimate weights of waste material parameters. In cases where bags are not opened, a brief written description of the contents of the bags must contain an estimate of the amount of each waste type in the bags. The written records of visual examination must be supplemented with the audio/video recording.

C4-4 Sample Custody of Samples

Chain-of-Custody on field samples (including field QC samples) will be initiated immediately after sample collection or preparation. Sample custody will be maintained until the associated analyses are completed and the data have been validated at the project level. Sample custody will be maintained until the sample is expended or until the sample is removed from the sample analysis program. Site QAPjPs will include a copy of the sample chain-of-custody form; this form will include provisions for each of the following:

- Signature of individual initiating custody control, along with the date and time

- Documentation of sample numbers for each sample under custody

- Signatures of custodians relinquishing and receiving custody, along with date and time of the transfer

- Description of final waste container disposition, along with signature of individual removing waste container from custody

- Comment section

C4-5 Sample Packing and Shipping

In the event that the analytical facilities are not at the generator site, the samples must be packaged and shipped to an off-site laboratory. Sample containers must be packed to prevent any damage to the sampling container and maintain the preservation temperature, if necessary. Department of Transportation (DOT) regulations must be adhered to for shipment of the package.

When preparing SUMMA® canisters for shipment, special care must be taken with the pressure gauge and the associated connections. Metal boxes which have separate compartments, or cardboard boxes with foam inserts are standard shipping containers. The chosen shipping container may be required to meet selected DOT regulations. If temperatures must be maintained, cold packs can be added to the package.

Glass jars are wrapped in bubble wrap or another type of protection. The wrapped jar should be placed in a plastic bag inside of the shipping container, so that if the jar breaks, the inside of the shipping container and the other samples will not be contaminated. The plastic bag will enable the receiving analytical lab to prevent contamination of their shipping and receiving area. Plastic jars do not present a problem for shipping purposes. A DOT approved cooler, or similar package may be used as the shipping container. If temperatures must be maintained, cold packs can be added to the package. If a fill material is needed, compatibility between the samples and the fill should be considered.

Sample containers should be affixed with a tamper-proof seal so that it is apparent if the sample integrity has been compromised. A seal should also be placed on the outside of the shipping container for the same reason. Sample custody documentation must be placed inside of the shipping container, with the current custodian signing to release custody. The shipping documentation will serve as proof of custody during shipment, so the transporter does not need to sign the chain-of-custody documentation.

A Uniform Hazardous Waste Manifest is not required, since samples are exempted from the definition of hazardous waste. All other shipping documentation (i.e., bill of lading, site-specific shipping documentation) is required.

C4-6 List of References

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1 **TABLES**

TABLE C4-1

GAS SAMPLE CONTAINERS AND HOLDING TIMES

Parameter	Container	Minimum Drum Headspace Sample Volume[a]	Holding Temperatures	Field Holding Time[b]	Shipping Allowance	Laboratory Holding Time[c]
VOCs	SUMMA® Canister	250 ml	0-40 °C	4 days	2 days	28 days

[a] Alternatively, if available headspace is limited, a single 100 ml sample may be collected for determination of VOCs.
[b] From the time of headspace sample collection to shipment.
[c] Programmatic-based maximum holding time. Holding time begins at VTSR.

TABLE C4-2
SUMMARY OF DRUM FIELD QC HEADSPACE SAMPLE FREQUENCIES

QC Samples	Manifold	Direct Canister	On-Line Systems
Field blanks[a]	1 per sampling batch[d]	1 per sampling batch[d]	1 per on-line batch[f]
Equipment blanks[b]	1 per sampling batch[d]	once[e]	1 per on-line batch[f]
Field reference standards[c]	1 per sampling batch[d]	once[e]	1 per on-line batch[f]
Field duplicates	1 per sampling batch[d]	1 per sampling batch[d]	1 per on-line batch[f]

[a]Analysis of field blanks for VOCs (Table C8-2 of Appendix C8), only, is required. For on-line integrated sampling/analysis systems, if field blank results meet the acceptance criterion, a separate on-line blank is not required.

[b]One equipment blank or on-line sample must be collected, analyzed for VOCs (Table C8-2), and demonstrated clean prior to first use of the headspace gas sampling equipment with each of the sampling heads, then at the specified frequency, for VOCs only thereafter. Daily, prior to work, the sampling manifold, if in use, must be verified clean using an OVA.

[c]One field reference standard or on-line control sample must be collected, analyzed, and demonstrated to meet the QAOs specified in Appendix C8 prior to first use, then at the specified frequency thereafter.

[d]A sampling batch is a suite of samples collected consecutively using the same sampling equipment within a specific time period. A sampling batch can be up to 20 samples (excluding field QC samples), all of which must be collected within 14 days of the first sample in the batch.

[e]One equipment blank and field reference standard must be collected after equipment purchase, cleaning, and assembly.

[f]An on-line batch is the number of samples collected and analyzed within a 12-hour period using the same on-line integrated sampling/analysis system.

TABLE C4-3

SUMMARY OF SAMPLING QUALITY CONTROL SAMPLE ACCEPTANCE CRITERIA

QC Sample	Acceptance Criteria	Corrective Action[a]
Field blanks	VOC amounts < 3 x MDLs in Table C8-2 of Appendix C8 for GC/MS and GC/FID; < PRQLs in Table C8-2 for FTIRS	Nonconformance if any VOC amount > 3 x MDLs in Table C8-2 of Appendix C8 for GC/MS and GC/FID; > PRQLs in Table C8-2 for FTIRS
Equipment blanks	VOC amounts < 3 x MDLs in Table C8-2 of Appendix C8 for GC/MS and GC/FID; < PRQLs in Table C8-2 for FTIRS	Nonconformance if any analyte amount > 3 x MDLs in Table C8-2 of Appendix C8 for GC/MS and GC/FID; > PRQLs in Table C8-2 for FTIRS
Field reference standards or on-line control sample	70 - 130 %R	Nonconformance if %R < 70 or > 130
Field duplicates or on-line duplicate	RPD ≤ 25	Nonconformance if RPD > 25

[a]Corrective action is only required if the final reported QC sample results do not meet the acceptance criteria.
MDL = Method detection limit
%R = Percent recovery
RPD = Relative percent difference

TABLE C4-4

SAMPLE HANDLING REQUIREMENTS FOR HOMOGENEOUS SOLIDS AND SOIL/GRAVEL

Parameter	Suggested Quantity[a]	Required Preservative	Suggested Container	Maximum Holding Time[b]
VOCs	15 grams	Cool to 4°C	Glass Vial[c]	14 Days Prep/ 40 Days Analyze[d]
SVOCs	50 grams	Cool to 4°C	Glass Jar[e]	14 Days Prep/ 40 Days Analyze[d]
Polychlorinated Biphenyls (PCBs)	50 grams	Cool to 4°C	Glass Jar[e]	14 Days Prep/ 40 Days Analyze[d]
Metals	10 grams	Cool to 4°C	Plastic Jar[g]	180 Days[h]

[a]Quantity may be increased or decreased according to the requirements of the analytical laboratory, as long as the QAOs are met.
[b]Holding time begins at sample collection (holding times are consistent with SW-846 requirements).
[c]VOA vial, must have septum cap.
[d]40-day holding time allowable only for methanol extract - 14-day holding time for non-extracted VOCs.
[e]Opaque glass container, must have Teflon® lined cap (example, amber jar).
[f]Analysis for PCBs is required only for waste streams in matrix parameter category S3220 (organics sludges).
[g]Polyethylene or polypropylene preferred, glass jar is allowable.
[h]Holding time for mercury analysis is 28 days.

FIGURES

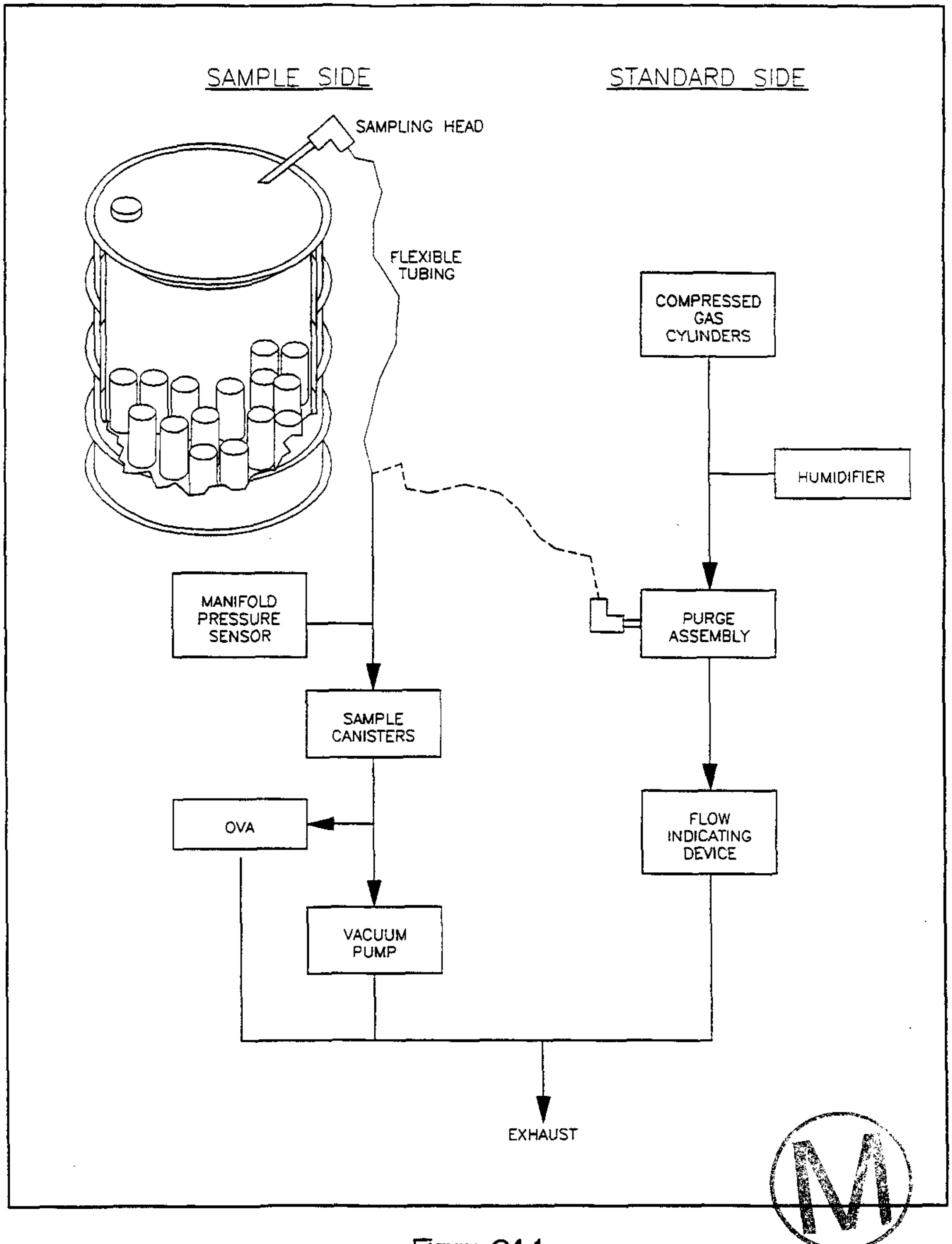


Figure C4-1
Headspace Sampling Manifold

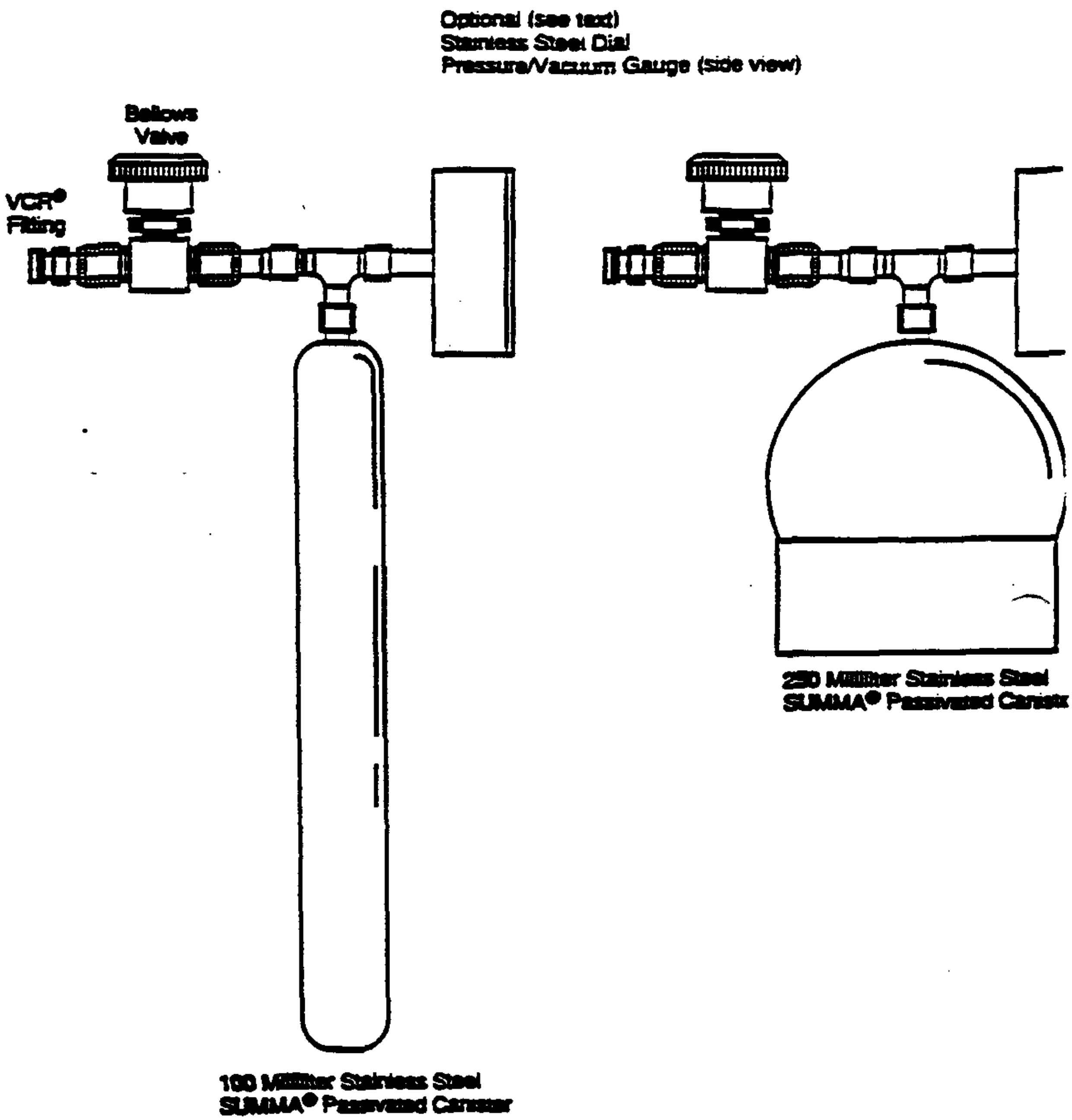


FIGURE C4-2

SUMMA® Canister Components Configuration

(Not to Scale)

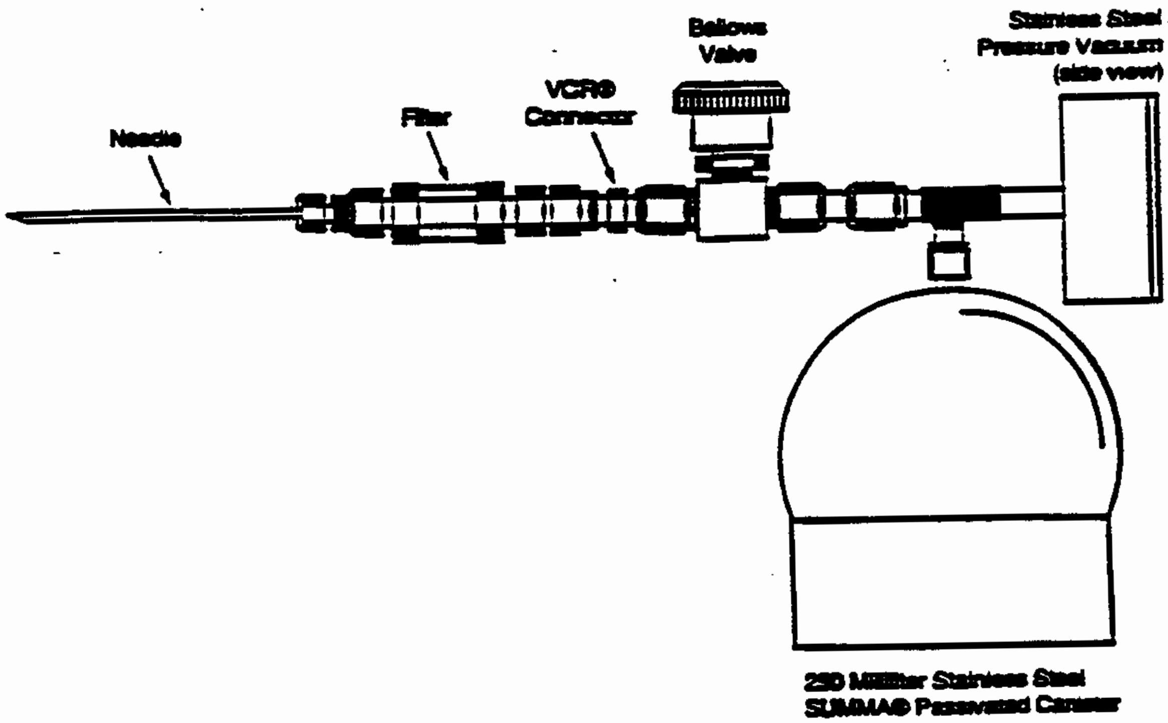
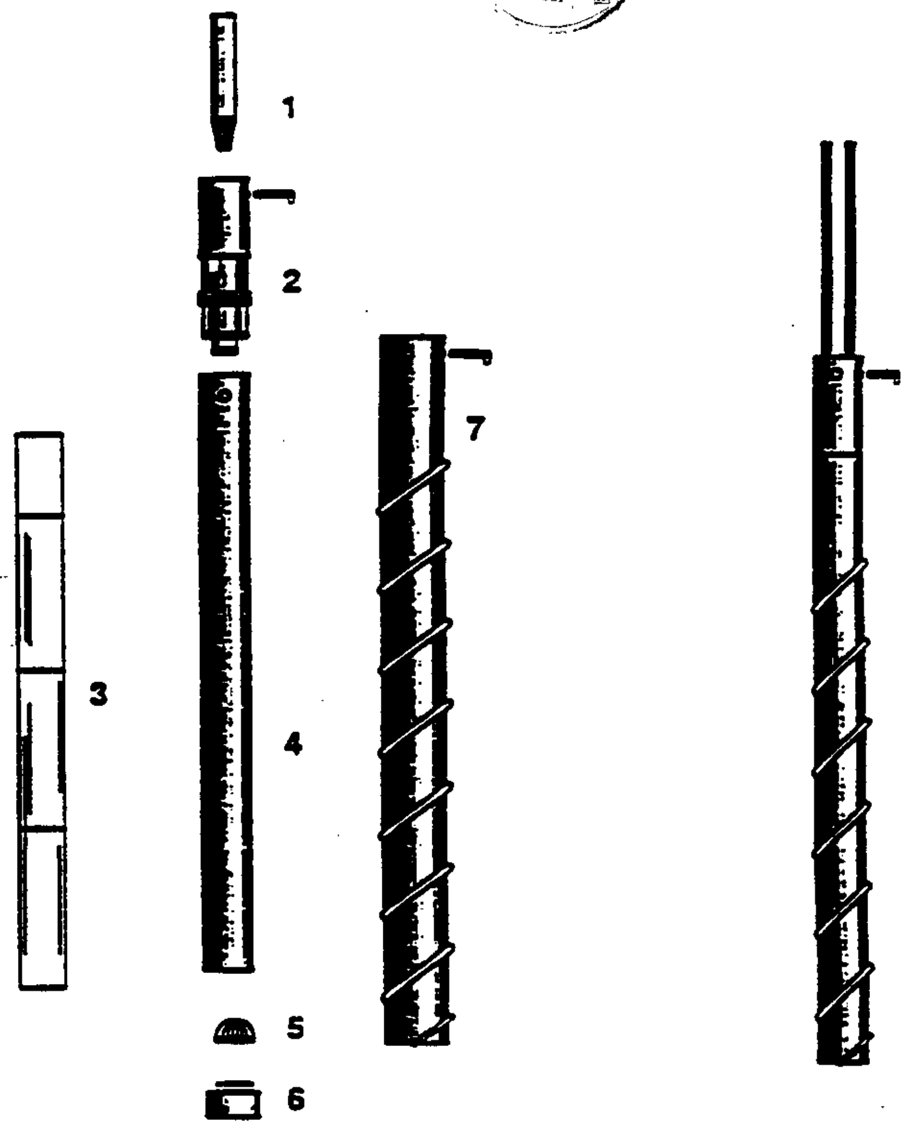


FIGURE C4-3

Schematic Diagram of Direct Canister with the Poly Bag Sampling Head



1 Drill Rod
2 Thrust Bearing Ball Check Valve
3 Clear Teflon® Liners
4 Core Barrel
5 Spring Retainer (optional)
6 Core Barrel Tip
7 Auger and Pin

FIGURE C4-4
Rotational Coring Tool
(Light Weight Auger)

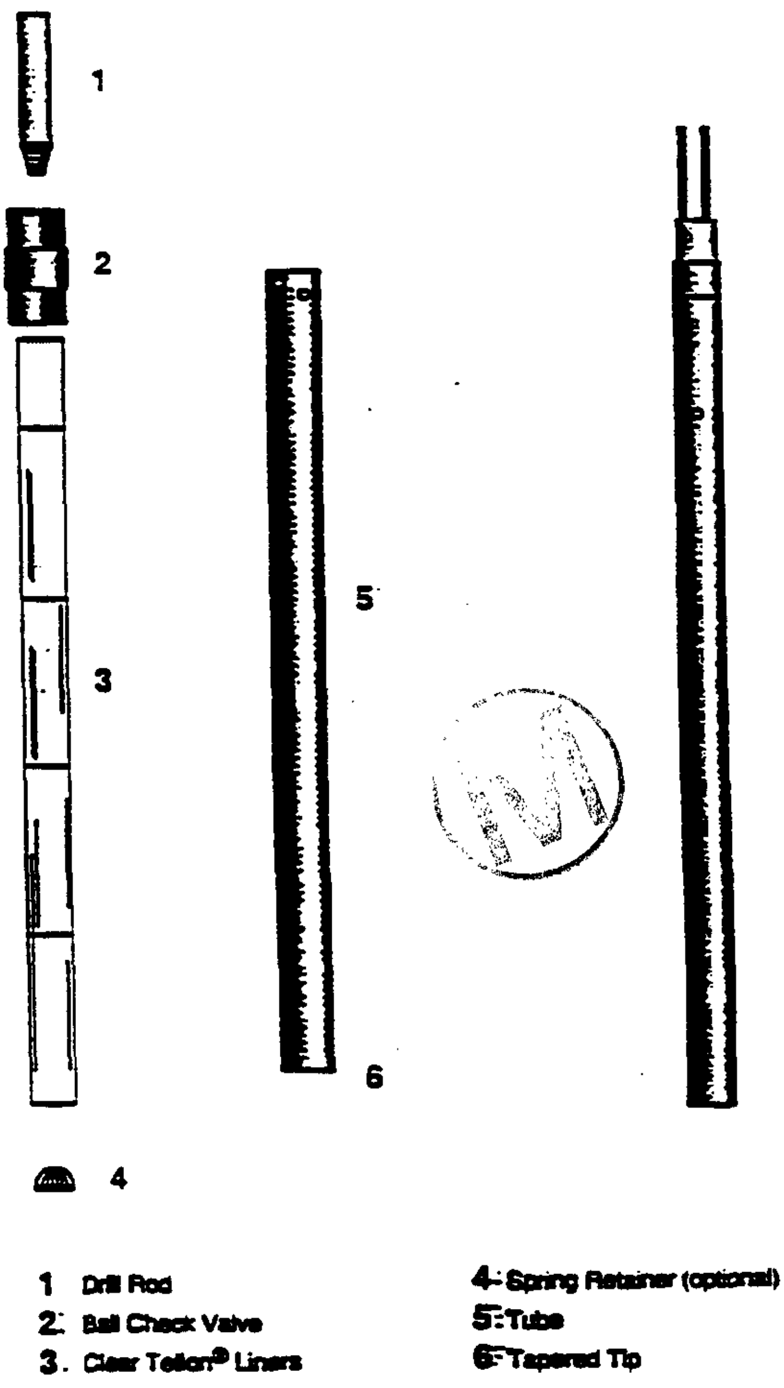


FIGURE C4-5
Non-Rotational Coring Tool
(Thin Walled Sampler)

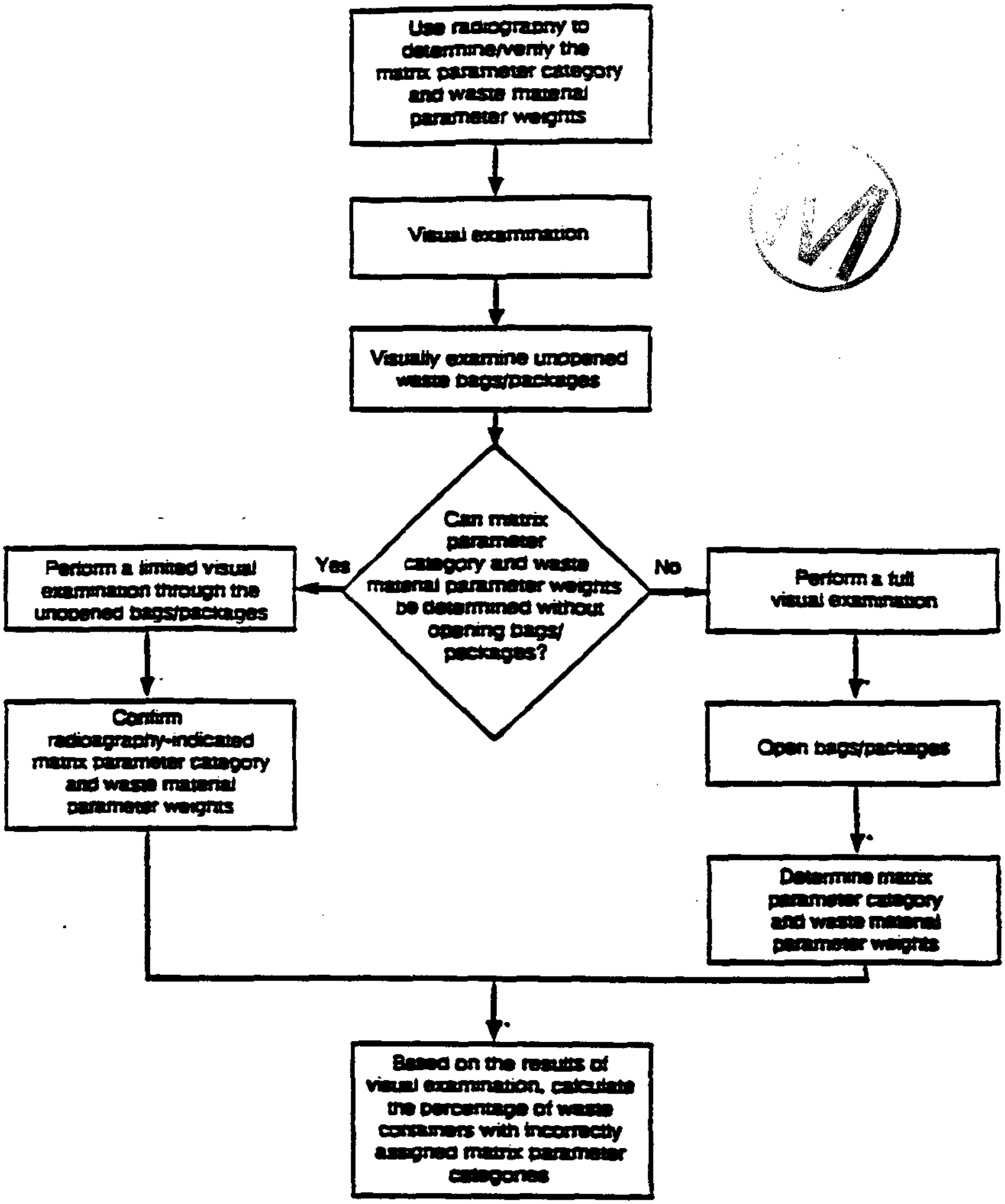


FIGURE C4-6

Overall Programmatic Approach to Visual Examination